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Short Communication

A METHOD FOR THE DETERMINATION OF GRAPHITIC CARBON IN GRAPHITE ORES

A. A. Qureshi, Khadim Hussain and Abdul Hafeez

PCSIR Laboratories, Lahore 16

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Many workers[1-4] have carried out the conversion of graphite into graphitic acid by oxidation with the help of a mixture of conc H_2SO_4, HNO_3 and $KClO_3$. Hummers and Offeman[5] used an essentially anhydrous mixture of H_2SO_4, $NaNO_3$ and $KMnO_4$ for the preparation of graphitic acid. The product obtained is washed till neutral, dried to constant weight and the percentage of graphite calculated from the formula $C_{28}H_{10}O_{15}$ which represents graphitic acid.[3] Montagut and Mensa[6] boiled a mixture of graphite containing amorphous carbon, conc H_2SO_4 and K_2SO_4 in a Kjeldahls flask for 2.5 hr. The mixture was cooled, diluted with distilled water, filtered, and the solid residue was ignited in oxygen. The loss gave graphitic carbon. Majamdar[1] heated a sample of adulterated graphite for 5–20 min at 700°C in a porcelain dish. The sample after cooling and weighing was again heated at 900°C. The loss was again recorded. The difference was due to graphitic carbon with ± 2% of error.

The present method is based on the difference in oxidation temperature of graphite and amorphous carbon which according to Partington[8] is about 370°C.

Table 1

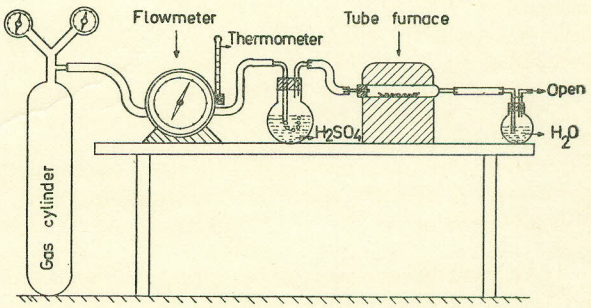
Temp °C	Time hr	Loss due to charaoal %	Rate of O_2 ml/min
400	1	46.9	272
400	2	49.2	272
450	1	48.1	264
450	1	48.80	268
450	1	49.50	272
450	1	50.30	300
450	1	51.00	308
450	1	49.40	272
450	1	49.70	272
450	1	49.55	272
500	1	51.55	272

Experimental

An intimate mixture of 0.25 g each of pure graphite and pure charcoal (ash 0.7%) was weighed in a stainless steel boat and introduced into the tube of the tube furnace. Pure dried oxygen was passed over the mixture. The experiments were conducted at various temperatures, for different time periods and with different oxygen flow rates. Every time the loss in weight of the mixture was recorded which was due to charcoal. The effect of time, temperature, the rate and quantity of oxygen flow has been experimentally determined (Table 1).

Results and Discussion

According to Partington[8] amorphous carbon begins to oxidise at 200°C and graphite at 570°C



Fig, 1

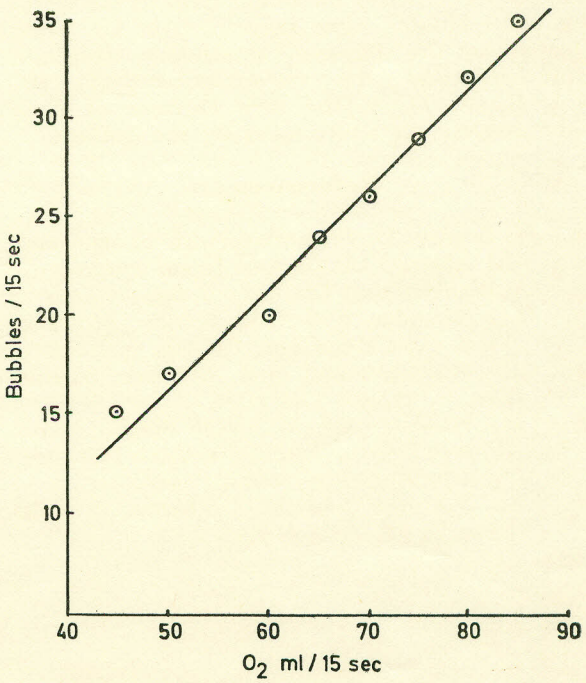


Fig. 2

but the rate of oxidation at these temperatures are very slow. It has been observed that even at 400°C and for 1 hr, the oxidation of charcoal is incomplete at an oxygen flow rate of 272–300 ml/min. (This oxygen rate having been experimentally determined to be optimum). It has also been observed that, for the same time and O_2 flow rate as above, if the temperature is raised to 500°C, graphite also begins to oxidise. This oxidation at lower temperature may be attributed to the presence of oxides in graphite ores. The ash content of the charcoal may as well be responsible for initiating the oxidation of graphites.

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